

Rapid On-Site Methods of Chemical Analysis

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by

R.E. Montgomery
IT Corporation
Edison, New Jersey
and
David P. Remeta
Michael Gruenfeld
U.S. Environmental Protection Agency
Risk Reduction Engineering Laboratory
Edison, New Jersey

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Project Officer

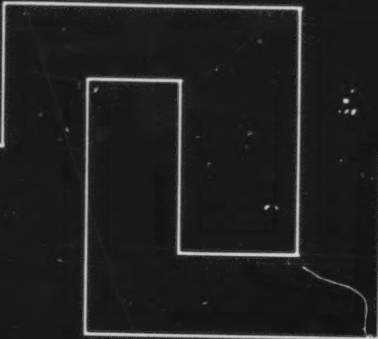
David P. Remeta
Superfund Technology Demonstration Division
Risk Reduction Engineering Laboratory
Cincinnati, Ohio 45268

RISK REDUCTION ENGINEERING LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

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RAPID ON-SITE METHODS OF CHEMICAL ANALYSIS

R E Montgomery*, D P Remeta** and M Gruenfeld**

* IT Corporation

** US Environmental Protection Agency

1 INTRODUCTION

The analysis of potentially hazardous air, water and soil samples collected and shipped to service laboratories off-site is time consuming and expensive. This Chapter addresses the practical alternative of performing the requisite analytical services on-site. The most significant application of such methodology is the initial appraisal of chemical waste sites and identification of highly contaminated areas. Hence, the majority of the methods reviewed are selective for classes of compounds, providing semi-quantitative and/or semi-qualitative information. These methods are useful as screening techniques for monitoring the general levels of contamination in a specific area.

The major advantages of field-oriented chemical analysis methods are real time sample analyses and reduced costs. Since field methods are less intricate than conventional methodology, sample throughput is increased substantially, resulting in cost savings. Costs are further reduced through the use of portable instruments that are relatively inexpensive to operate and maintain.

The use of rapid on-site methodology does not preclude further characterisation of samples for the presence of specific toxicants. In this regard, field methods may assist in identifying certain key analytical parameters which are then examined at a laboratory facility equipped with the appropriate instrumentation.

As a final consideration, there is a delicate balance between rapid on-site measurements and the need for legal/technical defensibility. In most instances, rapid on-site techniques cannot

furnish data of sufficient accuracy for litigation purposes and will have to be augmented by laboratory-based analyses. The use of mobile analytical laboratories (as described in Section 9) may be a practical alternative in such situations.

2 SCOPE

This Chapter addresses the problem of monitoring contaminated land for the presence of hazardous and/or toxic materials. It is primarily concerned with methods of chemical analysis that can be readily applied in the field, can provide real time data and be cost effective. The study has been restricted to a user survey rather than an extensive literature search. A user survey supplies information on available techniques and provides recommendations regarding their proper application.

The chemical analysis methods reviewed in this Chapter are presently used for emergency response to hazardous material spills and uncontrolled hazardous waste sites in the United States. Most of the techniques are interim in nature and are employed in the field by several components of the US Environmental Protection Agency (US EPA)* and their private contractors. These rapid chemical analysis methods vary in complexity, ranging from non-specific semi-quantitative screening techniques to compound specific, quantitative, multi-parametric analytical protocols. The techniques are described briefly and evaluated in the context of adaptation to field situations. Owing to the limited scope of the survey a large number of potentially useable techniques that were identified were not evaluated but are listed in Tables 11.8 to 11.10. This Chapter therefore provides a compendium of field methods and analytical techniques, and provides a critique of their practicality and utility for the rapid on-site assessment of contaminated land.

3 SAMPLING

Evaluation of the type(s) of contaminant(s) and extent of contamination at a particular site is dependent on both the availability of appropriate analytical techniques and the sampling design employed at the site. The sampling design will ultimately determine the number of samples to be collected and analysed, and the matrix and/or matrices to be sampled. Sampling designs will necessarily vary for each site. The extent and degree of contamination may be more obvious at certain sites, allowing a truncated sampling program. At other sites, contamination may be more diffuse requiring more extensive sampling and analysis.

* Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

3.1 Sampling Design

A comprehensive survey of a contaminated area involves the thorough characterisation of the magnitude and distribution of chemical contamination. However, investigation of sites can rarely examine the whole site in detail. Therefore, an adequate evaluation is largely dependent on the sampling pattern selected. Two principal types of pattern are routinely applied to the investigation of contaminated sites:

- (i) non-systematic techniques (eg, simple random sampling);
- (ii) systematic methods (eg, regular sampling grids).

Grid sampling (as opposed to simple random sampling) generally produces a more even coverage of the site and thereby provides a more reliable estimate of the spatial distribution of contaminants over the area. In both cases, the fixing of sample locations should enable the position of trial pits, boreholes, sampling sites, etc, to be easily identified during site investigations and during and after site cleanup. Sampling should be carefully designed to suit the particular needs of each site.

The sophistication of the sampling design and collection method(s) will depend on the size of the impacted area, the extent of contamination, the matrix being sampled, and the nature of the suspect chemical contaminant(s). For most sites, the number of sampling points should be sufficient to define the contaminated area. The lateral and vertical distributions of contaminants at many sites are related to the former use(s) of different parts of the site. The intensity of contamination may vary between different sites of similar history, but in general their patterns of contamination will exhibit similarities. Such relationships are likely at industrial sites with basic designs conforming to established practice. Other types of sites may be of an intermediate character (eg, scrap yards, industrial premises). Where such relationships exist and the site history is known, it may be possible to make use of this information in the sampling design.

Determination of what constitutes adequate sample numbers is more difficult. Preliminary estimates of the numbers of sampling points required can be obtained by the following methods:

- (i) Establishing a systematic sampling plan and adjusting the sampling frequency to suit the area to be investigated. This method provides a minimum number based on the dimensions of the site. On very large sites, this minimum number may mean that large areas of the site are not represented by a sampling point.

- (ii) Adjusting the sampling density based upon consideration of information on the former uses of individual parts of the site. Allowance is made for the expected pattern of the contamination, which may be non-uniform. In the remaining areas of the site the density of sampling may not be adequate.
- (iii) Specifying the desired probability of finding isolated areas of contamination of given size within the site under investigation.

A brief discussion of sampling design patterns and determination of adequate sample numbers based on work¹ in the UK is presented in Appendix H. The US, EPA has published a document giving sampling procedures, methods, materials, etc as part of a manual on the specific requirements of waste site and hazardous spill investigations.²

3.2 Sample Matrices

Determination of the distribution of contamination involves both sample collection and sample analysis. The sophistication of the sample collection method depends on the matrix or media being sampled and the substances of interest. The choice of practical analytical techniques is based on the nature of the class or classes of compounds being determined rather than on the sample matrix. Rapid on-site analytical methodology must successfully deal with the analysis of hazardous substances extracted from a variety of matrices including air, soil/sediment, and water, as well as concentrated wastes from chemical storage containers (eg drums, tanks, lagoons).

Methods for the analysis of aqueous samples are more well developed than those currently available for analysing air, soil and chemical wastes.³ This is partially due to technological advances in water quality programs among the NATO/ CCMS member nations. Consequently, small sample volumes are usually sufficient for analysis. Sampling of water becomes more complicated when insoluble or immiscible substances are present and form a separate phase or layer in the water column. In such instances, solvent extraction of sample composites followed by chromatographic analysis of the extracts yields rapid qualitative and quantitative information. Caution should be exercised when sampling well waters to assess groundwater contamination. Lee and Jones⁴ reviewed and recommended guidelines for sampling groundwater, noting that well water composition was frequently a function of the extent to which the well was pumped prior to sampling, the length of time since water was last withdrawn from the well, and the sampling methods.

The analysis of soil samples is more difficult than analysis of aqueous samples. The heterogeneous nature of sediment and soils makes it difficult to obtain a homogeneous sample. Analysis of soil and sediment samples involves an extraction procedure to bring the contaminant(s) into solution. These extracts often require extensive cleanup steps to remove substances interfering with the analysis. Several rapid chromatographic and spectroscopic methods are available for the analysis of soil samples for priority pollutants (see Section 9.1).

Analysis of chemical storage container contents on-site is especially important to facilitate segregation of potentially reactive and incompatible hazardous wastes. Compatibility testing of uncharacterised wastes is an integral component of uncontrolled hazardous waste site cleanup protocols. Section 4 describes several approaches for characterising concentrated wastes by using a chemical compatibility classification scheme. These field tests provide a rapid qualitative and semi-quantitative profile of the chemical waste(s) present. A more quantitative method for analysis of chemically compatible wastes involves sample compositing, and characterisation via multi-parametric analytical instrumentation provided by mobile laboratories operating on-site. The nature and applicability of such methodology is reviewed in Section 9.

Air monitoring differs substantially from the sampling and analysis of soil, water, and/or concentrated waste. A primary concern at spills and contaminated sites is the collection of hazardous and potentially toxic vapours. Owing to the variability in the physico-chemical properties (ie vapour pressure, fugacity, diffusion, chemisorption, etc) of hazardous substances and the open atmosphere, air monitoring requires an array of sampling and analysis techniques. Highly volatile materials are usually detected through the use of organic vapour analysers and colorimetric detector tubes. Low-volatility substances often require composite sampling followed by desorption from the stationary phase into a gas analyser. Section 5 describes air monitoring techniques that are commercially available and routinely applied at contaminated sites (reference should also be made to Chapter 10).

3.3 Quality assurance

Quality assurance (QA) defines the acceptance limits of measurement and monitoring data in terms of sensitivity, reproducibility, detection limits, and accuracy. Quality assurance/quality control (QA/QC) protocols are employed to demonstrate the reliability of sample analysis data and to assure that such measurements are performed in strict compliance with all applicable QA requirements.⁶

Many of the rapid on-site analytical methods reviewed in this Chapter provide either qualitative or semi-quantitative information. Therefore, normal stringent quality assurance procedures may not always be applicable. Prior to use in the field, kits and field instruments should be calibrated to determine their reproducibility and stability under defined conditions using prepared single or mixed standards. Calibration standards may also be prepared for field use, and when warranted, calibration samples can be further evaluated from simulated sample matrices. Sections 5-8 of this Chapter describe field monitoring techniques and provide QA specifications for these instruments.

A routine QA program should monitor the age of the reagents used.³⁶ In some rapid analytical techniques, reagents are labile or have a limited shelf life (eg hygroscopic or biological reagents). The purity of reagents (including distilled water) should always be monitored to prevent introduction of contaminants and trace impurities during sample preparation. Many analytical procedures are subject to interference from substances which may be present in chemical waste samples. Whenever interference is encountered or suspected, the analyst must eliminate the interference through use of an appropriate sample cleanup technique. A qualitative estimate of the presence or absence of interfering substances in a particular determination may be made by means of a recovery procedure that involves applying the analytical method to:

- (i) a reagent blank;
- (ii) a series of known standards covering the expected range of concentration of the sample;
- (iii) the sample itself (analysed in duplicate); and
- (iv) the recovery samples (prepared by adding known quantities of the substance analysed to split portions of the sample).

Representative elements of a QA program for on-site monitoring of contaminated land are presented below. This list may be added to or modified in accordance with the specific analytical requirements of each particular site.

- (i) Sampling - Adequate records of sampling and sample numbers should be maintained. Sampling techniques should be compatible with the analytical technique(s). On occasion, it may be advisable to freeze duplicate 'archive' samples.
- (ii) Sample preparation - Is the sample homogeneous or heterogeneous? Is the sample representative? Will the sample preparation technique alter the chemical species to be analysed?

- (iii) Instruments and techniques - When appropriate, calibrate the procedure (for procedures in constant use, calibration should be performed routinely at defined intervals). When appropriate, determine the reproducibility of the measurement (ie use standards or split subsamples).

The Oil and Hazardous Materials Spills (OHMS) Branch of the US EPA has developed an on-site QA/QC program for use aboard mobile laboratories which is designed to permit rapid dissemination of analytical results. Several protocols of the QA/QC program are designed to generate validation data concurrently with sample analysis activities. These protocols provide sample analysis data of known quality for establishing specific QC guidelines. The simultaneous reporting of sample analysis and QA validation data furnishes analytical results in 'real time'. It is then possible to determine the need and practicality of repeated or supplementary analyses. This type of QA management system permits the immediate release of sample analysis results to those primarily responsible for implementing appropriate remedial techniques during environmental cleanup operations.

4 COMPATIBILITY TESTING

The myriad of chemicals in complex matrices commonly found at hazardous waste or other contaminated sites exclude most conventional methods used to characterise samples. Comprehensive identification and quantification of these materials is not feasible owing to the cost and time required for such analyses. However, owing to the nature of the hazards involved in sampling and analysing chemical wastes, it is important to ensure the safety of personnel involved in the monitoring and cleanup of contaminated sites. Therefore, rapid on-site compatibility tests are used to enable the safe, effective and economic use of available manpower and equipment.

4.1 Description

Compatibility tests are used throughout a contaminated site management program to facilitate:

- (i) the determination of safe proximate storage of wastes on site;
- (ii) compositing of samples for reduced comprehensive analyses;
- (iii) compositing of wastes for efficient disposal and removal from the site;

- (iv) the determination of safe transportation of hazardous materials; and
- (v) the determination (and often definition) of the ultimate disposal method.

Perhaps most importantly, compatibility tests decrease the possibility of creating a greater environmental hazard by indiscriminate mixing of incompatible wastes.

Determining the compatibility of waste materials is not limited to chemical analyses. In a survey⁸ of emergency response workers, 43% stated that no instrumentation was used to identify the hazardous material. Indirect methods, such as reading placards or shipping papers or calling CHEMTREC*, were used. Once the materials are sufficiently identified, the compatibility of the specific compounds needs to be determined. Hatayama et al.⁹ described a systematic method for determining the compatibility of wastes. Compounds are assigned to specific groups through extensive reference tables, and the compatibility of binary combinations is predicted using a hazardous wastes compatibility chart. In contrast, Turpin et al.¹⁰ and Mayhew et al.¹¹ offer compatibility schemes for mixtures of wastes rather than specific compounds within a mixture (see Figures 11.1-11.4 and Tables 11.1 and 11.2). Although the Turpin¹⁰ and Mayhew¹¹ methods are less precise, they are rapid and better suited for on-site use. These compatibility tests determine which wastes can be stored, combined and/or shipped together for more efficient removal, and the types of ultimate disposal options available.

Compatibility testing is multifaceted and is used in many phases of contaminated land remediation. This section focuses on specific on-site analytical methods that comprise the backbone of compatibility testing.

4.2 Selected Methods

The US EPA has numerous federal, regional and contractor support groups to assist in the characterisation of hazardous waste sites. One of these, the Environmental Response Team (ERT), was established

* CHEMTREC is an acronym for the Chemical Transportation Emergency Center, a 24-hour toll-free hotline that provides technical information regarding emergency evaluation and response to spills of chemicals and hazardous substances. CHEMTREC maintains a directory of industry experts and co-operatives that can be contacted to provide technical advice and/or on-scene assistance.¹²

Table 11.1 Turpin¹⁰ Compatibility Tests

Test	Category
1 pH*	Caustic (NF) Caustic (F) Acid (NF) Acid (F)
2 Water Reactive	
3 Oxidisation/Reduction	Oxidiser (F) Oxidiser (NF)
4 Radioactives	
5 Volatile vapour/gases	
6 Flammability	

* pH is the level at which the release of cyanide, sulphide and sulphide gases pose a threat
(F) Flammable
(NF) Non-flammable

Table 11.2 Selection Criteria for the Compatibility Field Testing Methods¹⁰

Category	High pH9	Low pH9*	HNU or OVA on vapour space	Flammability	Redox	R/A	B/T
A-Caustic (NF)	+	-	-	-	-	-	-
B-Caustic (F)	+	-	+	+	-	-	+
C-Acid (NF)	-	+	-	-	-	-	-
D-Acid (F)	-	+	+	+	-	-	+
E-Water Reactive	-	-	-	-	-	-	+
F-Oxidisers (F)	-	-	+	+	+	-	-
G-Oxidisers (NF)	-	-	-	-	+	-	-
H-Radioactives	-	-	-	-	-	+	-

* At pH9 the release of cyanide, sulphide and sulphide gases pose a threat

(F) Flammable R/A Radioactivity

(NF) Non-flammable B/T Beaker test for water reactivity

in 1978 to provide technical assistance to multimedia environmental problems. From 1979 to 1982, this group responded to more than 170 environmental emergencies and hazardous waste sites in the USA.¹³ Turpin et al¹⁰ have described six rapid on-site compatibility tests (see Table 11.1) that are routinely employed to assign waste drums into one of the eight categories. Most of the methods used by Turpin et al¹⁰ are described in the following sections. These methods are rapid, inexpensive and based on user experience. Table 11.3 lists these and additional compatibility methods often used on site. These methods vary greatly in expense and complexity, ranging from gas chromatographic analysis of PCBs to measuring temperature changes with a thermometer. The specific character of the hazardous waste site will determine which are the most appropriate in any particular case. Some of the most rapid and essential compatibility tests are reviewed below.

4.2.1 Radiation. A test for radiation is one of the first tests performed to ensure worker safety and to assist in the determination of ultimate disposal options. In the compatibility schemes reviewed (eg Figures 11.1-11.3), waste is initially scanned for radioactivity and isolated from the mainstream if necessary. Normal background gamma radiation is approximately 0.01 to 0.02 milliroentgen per hour (mR/hr) on a gamma survey instrument, and employee exposure should not exceed 10 mR/hr without the advice of a health physicist.¹⁰

4.2.2 Flammability. Although a 'SetaTM' flash closed cup flame tester allows an accurate determination of the degree of flammability, it is an impractical technique for monitoring large numbers of samples. Turpin et al¹⁰ have described a rapid method of determining flammability in which a beaker with 2-5 millilitres of sample is placed in a sandbox and a propane torch is slowly passed over the sample. The liquid is considered flammable if a flame is observed, and non-flammable if none is observed after several passes of the torch.

4.2.3 pH. The pH of a waste solution affects corrosivity and can initiate the release of hydrogen sulphide, hydrogen cyanide gas, ammonia gas or amine vapours. Colorimetric and electrochemical methods have been used to determine pH, but these methods are time consuming and inaccurate with dirty samples. The method selected by Turpin et al¹⁰ uses a multiband pH paper strip which contains a reaction zone for colour changes and a zone of reference colours. Any excess material on the strip is removed by wiping on a clean surface to reduce interferences from viscous and coloured samples.

4.2.4 Water reactivity. This rapid qualitative test provides a great deal of information. Small volumes of liquid are added successively to distilled water in a heavy walled glass test tube. One can then monitor whether heat is generated, if a gas or a

Table 11.3 Compatibility Tests

	Method	Type of Test	Deliverables
1	Water reactivity and solubility	qualitative	T,G,P miscibility, density (1, 1)
2	Karl Fischer analysis for water	semi-quantitative	Z water
3	pH	semi-quantitative	pH
4	Cyanide	qualitative	+/- cyanide
5	Sulphide	qualitative	+/- sulphide
6	Organic halogen	semi-quantitative	Z halides
7	PCBs by gas chromatography	semi-quantitative	50 mg/l, 50 mg/l, or ND
8	Compatibility of liquid wastes	qualitative	T,G,P miscibility, order of addition
9	Oxidising agents	semi-quantitative	active oxygen (mg/l)
10	Beilstein's test for halogens	qualitative	+/- halogens
11	Flammability	qualitative	+/- flammability
12	Radiation	quantitative	radioactivity
13	Volatiles	semi-quantitative	see HNU or OVA sections
14	Explosion potential	semi-quantitative	degree of explosion potential

Key: + = present
 - = absent
 ND = not detected

Z = w/v percent
 P = precipitate
 G = gas formation
 T = temperature

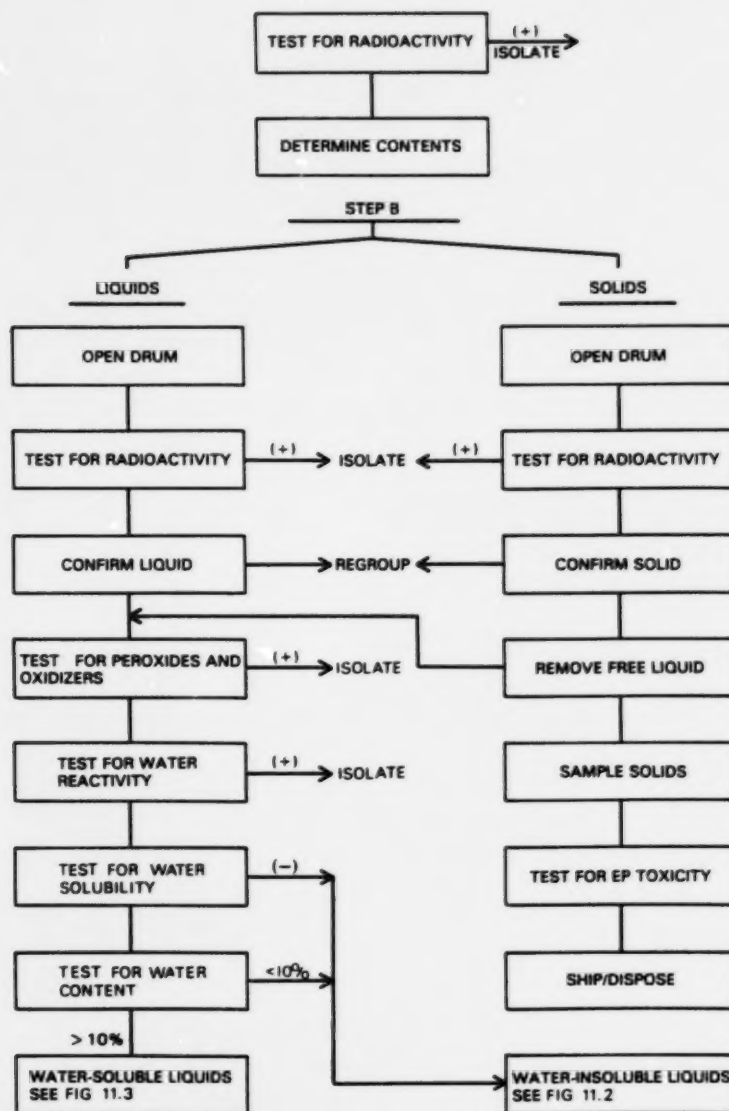


Figure 11.1 Compatibility Testing - Liquid/Solid Decision Tree (after Mayhew et al¹¹)

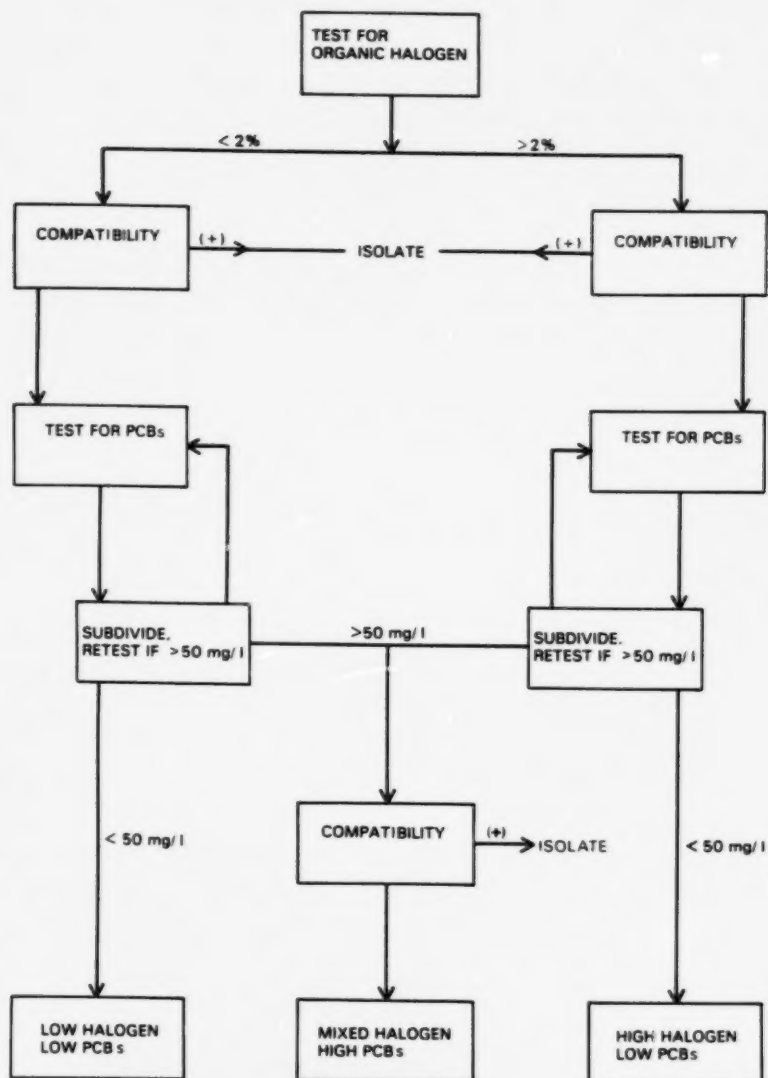


Figure 11.2 Compatibility Testing - Water Insoluble Liquids Decision Tree (after Mayhew et al¹¹)

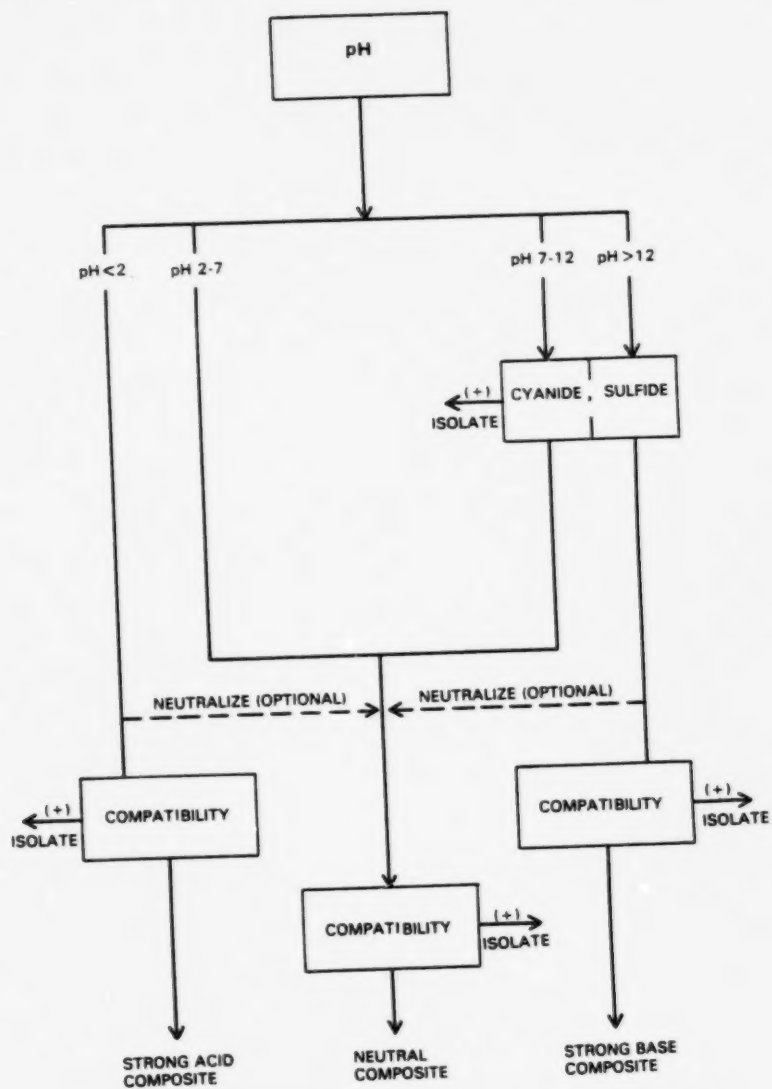


Figure 11.3 Compatibility Testing - Soluble Liquids Decision Tree (after Mayhew et al¹¹)

precipitate forms, if the waste is miscible with water, and the density of the waste (greater than or less than 1 gm/ml).

4.2.5 Beilstein's test. The combination of sensitivity and simplicity makes this a suitable scanning method for halogens in waste. A loop of copper wire is heated in a Bunsen burner until the flame is no longer coloured. The wire is cooled, dipped into the waste, and then heated in the Bunsen burner (a green flame indicates the presence of halogens). One drawback to this test is that volatile halogenated compounds may evaporate before the wire is sufficiently heated, causing an erroneous result. The sensitivity of the method is another limitation in that it is susceptible to interferences when 'dirty' samples are analysed.

4.2.6 Small batch sampling. This is probably one of the most practical and informative compatibility tests performed before drums are combined for disposal. Five to ten drum samples of 10 to 15 millilitres each are combined and the order of addition recorded. After each sample is added, the change in temperature, any precipitation or gas formation, the viscosity, and the miscibility are noted. This batch sample may then be analysed by gas chromatography for specific compounds, such as polychlorinated biphenyls.

4.2.7 Oxidation - reduction potentials. The hazards associated with the indiscriminate mixing of oxidisers and reducers make this an important initial test in characterising chemical wastes. In a joint venture with a private laboratory the US EPA recently developed a portable REDOX Field Test Kit for measuring oxidation-reduction potentials of chemical wastes.^{10,36} The kit consists of a battery operated pH meter capable of monitoring EMF changes, a platinum sensing combination electrode with a silver-silver chloride reference electrode, a test solution of 0.001 N ferrous ammonium sulphate, and a test solution of 0.001 N potassium chromate. A sample of the chemical waste is added to each of the test solutions. A change of 50 mV in the positive direction from the standard reading of 380 mV for the ferrous ammonium sulphate solution, indicates the presence of oxidisers. A change of 50 mV in the negative direction from the standard reading of 630 mV for the potassium chromate solution indicates that reducing species are present.

4.3 Application

Compatibility tests have only recently become widely used. The need for comprehensive compatibility tests was demonstrated at the Chemical Control Corporation waste disposal site in Elizabeth, New Jersey (USA). In the midst of the cleanup operation 40 000 drums of hazardous waste ignited and exploded during a massive chemical fire.

Table 11.4 Waste Group Colour Codes and Corresponding Waste Properties (from Rittaler¹⁴)

Colour Code	Waste Properties
White	pH 7, non-flammable
Red	pH 7, torch test properties
Red/orange	pH 7, Seta flash positive
Blue	pH 7, non-flammable
Yellow	pH 7, torch test positive
Yellow/green	pH 7, Seta flash positive
Orange	Water reactive
Green	Oxidiser, torch test positive
Blue/green	Oxidiser, Seta flash positive
Blue/white	Oxidiser, non-flammable
Red/white	Radioactive

Across the United States, environmental service organisations have been implementing a variety of compatibility tests. Since each site has its unique problems of sample collection, weather conditions, ultimate disposal, and financing, a variety of compatibility schemes have been developed. Three compatibility schemes are described below.

4.3.1 Callahan Uncontrolled Hazardous Waste Site, St Louis, Missouri. In 1980, the Missouri Department of Natural Resources, US EPA Region VII, and Ecology and Environment Inc undertook a preliminary investigation of the Callahan abandoned hazardous waste site. Monitoring revealed incompatibility and safety tests were performed on-site as drums were excavated. The tests made included measurements for radiation, hydrogen cyanide and organic vapours (HNU Hazardous Waste Sector - see 5.2). Samples were analysed for water reactivity, pH, Redox potential and flammability by the open torch and SetaTM flash closed cup methods. The results of these analyses determined the category of the waste drums (see Table 11.4).

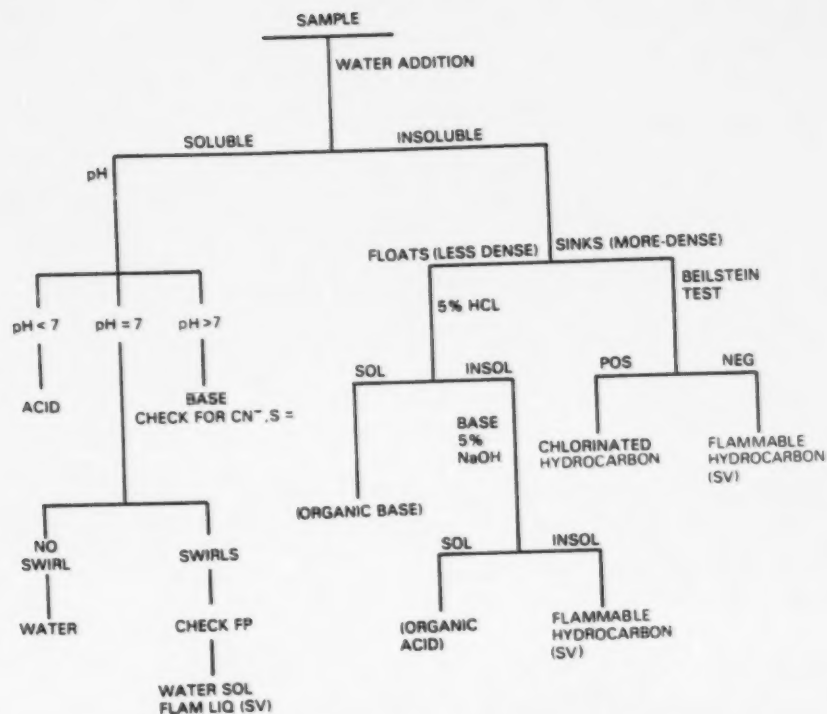
4.3.2 Picillo Farm, Coventry, Rhode Island. In 1978, the Rhode Island Department of Environmental Management initiated cleanup operations at this chemical dump site where solid, sludge and liquid wastes were discovered.¹⁵ Samples from the drums were transferred to a laboratory where any fuming or odour was noted and the liquid and sludge samples were tested for pH. Those with a pH less than 3 were considered acids, whereas those with a pH greater than 12 were considered basic. Following these tests, the flammability characteristics of the wastes were determined using a portable flash point tester. If the flash point was less than 60°C, the waste was

Table 11.5 Analytical Requirements for Disposal Applied at Picollo Farm (from Muller et al.¹⁵)

-
- | | |
|----|--|
| 1 | Flammability |
| 2 | pH |
| 3 | Specific gravity |
| 4 | PCB analysis |
| 5 | Thermal content (BTU/lb) |
| 6 | Physical state at 70°F |
| 7 | Phases (layering in liquids) |
| 8 | Solids (%) |
| 9 | Hydrocarbon composition |
| 10 | Pesticide analysis |
| 11 | Sulphur content |
| 12 | Phenols |
| 13 | Oil and grease (%) |
| 14 | Water (%) |
| 15 | Viscosity |
| 16 | Organochlorine-percentage |
| 17 | Metals analysis |
| | (a) Liquids were analysed for soluble metals |
| | (b) Solids were extracted according to the EPA Toxicant Extraction Procedure (14 hr) which shows leachate metals |
| | (c) Both liquid and solids were checked for concentration of the following metals: |
| | |
| | Arsenic Mercury |
| | Barium Nickel |
| | Cadmium Selenium |
| | Chromium Silver |
| | Copper Zinc |
| | Lead |
| 18 | Both free and total cyanide content were checked |
| 19 | Solids were checked for solubility in water, sulphuric acid and dimethyl sulphoxide |
-

considered flammable. Thereafter, a small amount of sample was added to distilled water to measure the water reactivity properties of the waste.

The samples were further examined for specific gravity and assigned to one of three groups with a specific gravity of: 0.9, 0.9 to 1.1, 1.1. Following the specific gravity test, all samples with a pH greater than 9 were checked for cyanide, and acids were tested for oxidation/reduction potentials to determine compatible groups



CODES USED FOR MARKING DRUMS:

- | | |
|-------------------------|----------------------------------|
| 1. ACID (pH =) | 7. FLAMMABLE LIQUID (SV) |
| 2. BASE CN FREE (pH =) | 8. ORGANIC ACID |
| 3. BASE DANGER CN | 9. CHLORINATED HYDROCARBON |
| 4. WATER (pH =) | 10. SOLID (MARK ON PART OF DRUM) |
| 5. WATER SOL SV | 11. OILS |
| 6. ORGANIC BASE | |

NOTE: OILS SEGREGATED AND CHECKED FOR PCB'S INDEPENDENTLY. EACH SOLVENT COMPOSITE (100 DRUM LOTS) CHECKED FOR PCB'S

Figure 11.4 Analytical Flow Chart Applied to Old Hardin County Brickyard, Kentucky (after Stroud et al³⁸)

for temporary storage of drummed wastes. Prior to disposal, the contents of 60 to 100 barrels determined to be compatible were composited, and batch samples from every five barrels were analysed for polychlorinated biphenyls. This was the most time consuming and costly analytical task in the compatibility testing scheme employed. Analyses performed for this waste site are listed in Table 11.5.

4.3.3 Old Hardin County Brickyard Hazardous Waste Site, West Point, Kentucky. Investigations started in 1971 by the Kentucky Department for Environmental Protection revealed approximately 3000 55-gallon (US) drums of liquid, sludge and solid wastes. In March 1982, cleanup operations commenced with on-scene personnel employing the analytical flow chart illustrated in Figure 11.4 to segregate the drums. The drums were assigned to one of eleven categories, and disposed of accordingly (eg waste-water treatment, solvent reclamation, incineration, landfilling, approved hazardous waste disposal facilities). When drums were labelled radioactive or explosive, experts were called upon to assist in hazardous assessment.

5 AIR MONITORING EQUIPMENT

Monitoring techniques and devices have been developed that survey ambient air, generating both qualitative and quantitative data.

The primary objectives of an air monitoring program are:

- (i) to determine the type and approximate quantity of airborne contaminants both on-site and in the vicinity of the site;
- (ii) to establish what level of personal protection should be used by on-site workers;
- (iii) to establish whether additional safety measures should be initiated for protection of the public, such as an evacuation plan; and,
- (iv) to provide a record of conditions that prevail during the course of any cleanup activities.

Air monitoring methods or techniques that are routinely and widely used comprise three basic categories:

- (i) methods that provide immediate results based on direct read-out from an analytical instrument or other indicator source;
- (ii) methods that allow for the collection of an air sample which must be analysed; and,
- (iii) passive detection devices that employ a chemical substance that reacts with the pollutant and changes its properties in some fashion.¹⁶

The following section reviews those techniques that have been evaluated and are currently in use by the US EPA.

5.1 Organic Vapour Analyser

5.1.1 Description. The Model 128 Century Organic Vapour Analyser (OVA) is a lightweight (5.5 kg) shoulder-borne gas chromatograph (GC) manufactured by the Foxboro Corporation of Burlington, Massachusetts. The OVA-GC is capable of monitoring volatile organic compounds in the air. It is equipped with a flame ionisation detector (FID) and uses an externally mounted GC column that enables separation of organic components under ambient temperature conditions. A portable temperature regulating accessory pack can be used for operating the unit isothermally. The GC column can be interchanged, permitting the use of different column lengths (225 mm, 300 mm, 600 mm) and packing materials.

The instrument can be used in either of two modes of operation, the total organic mode or the GC mode. In the total organic mode, a 'sniffer' device is used consisting of a hand-held output meter attached to a sample probe. The 'sniffer' device is interfaced to the main OVA module by an electrical cable. In the total organic mode, a 250 ul sample is semi-automatically introduced into the unit via the sample probe. The concentration (in ppm) of non-methane compounds in the air sample is then registered on the output meter. The total organic concentration response represents the sum of the percentage relative response for each organic constituent in the sample. A portable strip chart recorder is used to obtain a corresponding print-out of the total organic concentration response.

The GC mode of operation is used primarily for calibration of the instrument. This must be done prior to using the OVA in the total organic mode. In the GC mode, a sample is introduced into the unit via a gas-tight syringe injection through a septum. The portable strip chart recorder provides a printout of the response, as peaks similar to those obtained when using a laboratory scale GC. The output meter, which is used when the OVA is being operated in the total organic mode, can also be used in the GC mode along with the strip chart recorder. As a peak is printed out on the recorder, a total concentration response will register on the output meter. A maximum read-out (in ppm) on the output meter corresponds to the apex of the peak on the recorder.

Hydrogen gas serves as a fuel for detector combustion, and also as the carrier gas. A hydrogen supply tank is contained within the OVA-GC, and can be refilled from a master tank. The instrument can be operated using a battery, or a 120 V AC power supply. A battery charger is also part of the OVA-GC unit.

5.1.2 Evaluation. As part of an evaluation of the Model 128 OVA-GC conducted by the ERT of the US EPA the reproducibility of the following three instrument parameters was compared: peak height;

Table 11.6 Reproducibility of Three Instrument Parameters for 128 OVA-GC

Parameter	Coefficient of Variation Total Organic Mode	(Cv) GC mode
Peak height	5%	5%
Peak retention time	1-4%	1-3%
Relative retention time	5-10%	5-10%

peak retention time; and, relative retention time. The results of this evaluation are presented in Table 11.6. The detection limit of the unit is 0.2 ppm.

The OVA-GC does have limitations. It cannot detect inorganic compounds and air temperature fluctuations affect the operational performance¹⁹ in the field. Furthermore when monitoring ambient air in the total organic vapour mode, accurate readings are not obtained unless the 'sniffer' device is held or placed within inches of the contamination area(s).

5.1.3 Application. The OVA-GC has been used extensively by the ERT for the monitoring of air, soil and water for the extent of chemical contamination, and for the surveying of chemically contaminated lagoons and waste chemical drum storage sites for the identification of potential sampling points. It is used in preliminary field testing operations to determine safety measures to be taken and levels of personal protection to be used during on-site activities. The unit also serves as a method of evaluating the effectiveness of various cleanup operations.¹⁹

Abandoned Hazardous Waste Site, West Point, Kentucky. The ERT provided air monitoring and evaluation assistance at this site during January 1982. The area of concern was an abandoned hazardous waste site containing 1000 to 2000 drums of unidentified chemical waste. The initial stages of the air monitoring program utilised the Model 128 OVA operating in its total organic mode. A survey of the area was conducted using the OVA to fix locations from which air samples were subsequently collected. Air samples were taken from locations giving OVA total concentration readings ranging from 2 to 10 ppm using monitoring pumps equipped with Tenax thermal desorption tubes (see Section 5.4.2). Laboratory analysis of these samples revealed the presence of relatively low concentrations of toluene and xylenes.

Hauppauge, Long Island, New York. The OVA was used as part of an air monitoring program instituted by the ERT during November 1982 at an incident involving suspected contamination in a school. Samples were collected both on-site and at off-site stations established in the vicinity of the building using portable air monitoring pumps equipped with sorbent tubes packed with activated carbon and silica gel. The OVA was used in the total organic mode as a screening device to detect any 'hot spots' on the site, and to monitor areas other than the fixed sampling stations. Results of both the OVA screening and air sampling efforts revealed only low level contamination.

5.2 Hazardous Waste Detector

5.2.1 Description. The Model PI 101 Hazardous Waste Detector is a portable gas analyser manufactured by HNU Systems, Inc, Newton, Massachusetts. The instrument was designed to detect the presence of potentially harmful chemical vapours.

The Model PI 101 operates on the principle of photoionisation. Gases are drawn into a sample probe, and then into a lamp/ion chamber. Molecules are then ionised as a result of the absorption of ultraviolet light. This type of detection system is sensitive to vapours of both organic and inorganic composition.

The instrument is composed of two modules, the read-out unit and the sample probe, connected by an electrical cable. The read-out unit houses a DC rechargeable battery, an AC recharger and power supply, and an ion chamber. A direct concentration (ppm) linear scale read-out is located on the face of this module. The sample probe houses the lamp-ion chamber where photoionisation occurs, and a pump which transfers the gas sample from the probe to the ion chamber of the read-out unit. The sample probe also has a 300 mm tip extension.

The unit is sensitive to a wide range of compounds using three different sample probes. Each probe is equipped with a lamp having a different energy level (9.5 eV, 10.2 eV, 11.7 eV). Thus, suspect compounds are positively identified based on correlation of the known ionisation potential of the suspect compound through use of the appropriate probe.

The Model PI 101 is portable, having a total weight of less than 4.1 kg, and requiring no additional pumps or gas cylinders for operation. The unit can also be powered from 110 V AC, for use in the stationary mode. An optional portable recorder is available. The instrument is calibrated on benzene or vinyl chloride by the manufacturer, but can be calibrated on any compound as specified by the buyer.

5.2.2 Evaluation. Accuracy of the PI 101 is 96% at the 5 ppm concentration level, while instrument results are reproducible to $\pm 1\%$. The instrument is sensitive within the range of 0.1 to 2000 ppm.

The model PI 101 has several limitations in that it cannot be used to monitor for specific gases and vapours and it does not detect the presence of hydrogen, cyanide, or methane gases. The latter may be an advantage, however, when investigating municipal/industrial landfill sites where the methane produced can totally mask the presence of other more toxic vapours when a flame ionisation detector is used. An additional disadvantage is that the operational performance deteriorates in an uncontrolled environment since weather conditions (particularly increases in humidity) may limit the accuracy of results.

5.2.3 Application. Practical applications of the Model PI 101 include a broad range of situations because of the instrument's wide dynamic operating range. They include:

- (i) the detection of chemical leaks;
- (ii) the location of site 'hot spots' and below-ground level waste tanks and barrels;
- (iii) the determination of plume spread at hazardous material spill sites;
- (iv) the classification of work site hazard levels as an indication of personal protection requirements for workers at the site;
- (v) screening samples of soil, water, and other materials prior to laboratory analysis.¹⁹

Niagara Falls, New York. The Model PI 101 Hazardous Waste Analyser was used as an air monitoring device at a landfill site in Niagara Falls, New York. The instrument was used in the portable mode for the detection of 'hot spots' during an aquifer survey of the site perimeter in order to determine necessary safety precautions for on-site workers and to fix specific locations for air sampling.

5.3 Portable Photoionisation Gas Chromatograph

5.3.1 Description. The Photovac Inc 10A10 Portable Photoionisation Gas Chromatograph is designed for on-site monitoring low concentrations of volatile compounds in air.³⁷ The unit uses a photoionisation detector and air as the carrier gas. The gas chromatographic columns within the instrument operate at ambient temperature. Column lengths can range from 150 mm to 6 m, and

flexible capillary columns can be installed with adapters. Samples (1 μ l to 1 ml) are injected directly without any preconcentration. Two options are also available for the rapid screening of samples: a dual column arrangement and a carrier gas backflush valve.

The instrument weighs approximately 11 kg and is self-contained except for the recorder. The unit operates on rechargeable batteries or on 115 V AC. The air is supplied by a lecture bottle mounted on the top of the instrument which can deliver carrier gas for 10 days in typical operations.

5.3.2 Evaluation. The greater sensitivity of the Photovac 10A10 when compared with the HNU Model PI 101 makes it more appropriate in situations where volatile compounds in the low μ g/l concentrations are anticipated.

The photoionisation detector responds to a broad range of organics and inorganics such as phosphine, arsine, ammonia and nitric oxide. The two available options, dual column operation and backflush valves, make the 10A10 versatile. The dual column option allows for more precise identification of compounds when two columns of different packing materials are used, or for rapid screening and selective quantification when a long and a short column are used. The backflush valve routes the sample directly to the detector which records the total volatile content of the sample.

The instrument's linearity and sensitivity for specific compounds demonstrates its utility for on-site use. Hydrogen sulphide is detected at 0.1 ppb, with excellent linearity between 0.1 and 1 ppb and between 1 ppb and 5 ppb. In ambient air samples, where benzene was detected at approximately 4.5 ppb, the signal to noise ratio was 45. The coefficient of variation for benzene between 1 ppb and 1 ppm was less than 4%.

The limitations of the instrument are the power source, the carrier gas, and the ionisation potential of the detector. The batteries provide for 4 hours of operation and can be recharged while operating the instrument on AC. The lecture bottle of carrier gas lasts for approximately 24 hours of continuous operation. The photoionisation source uses photons of slightly less than 11 eV and should not detect molecules with a greater ionisation potential. In practice however, some molecules with a theoretical ionisation potential above this value are detected.²⁰

The instrument's primary limitation is operation of the gas chromatographic column at ambient temperature. Most volatiles can be detected at ambient temperatures. Higher boiling compounds (eg xylene, trichlorobenzene) can also be detected, but with reduced sensitivity. Ambient air columns are more prone to baseline drift and require additional stabilisation when the temperature changes.

This could occur when moving the instrument from a vehicle to the sampling area, or during daily temperature cycles. If high sensitivity (ppb) is required, the instrument requires a location free from temperature fluctuations with approximately 30 minutes to stabilise. At lower sensitivities (10 ppb to 1 ppm), acceptable performance can generally be achieved within 5 minutes.

5.3.3 Application. The Photovac 10A10 is most useful for screening air samples containing compounds in the low ug/l concentration. Typical situations can include the screening of volatiles emanating from drinking water downwind from the contamination source, tracking the front of a contamination plume, and ambient air monitoring. Since preconcentration is unnecessary, the instrument is quite portable.

Municipal Landfill. Monitoring of a municipal landfill in a large metropolitan area (since litigation is pending, the site shall remain unidentified) has been carried out. As parts of the landfill were filled, vent pipes were installed to provide venting of gas and vapours. Preliminary field testing determined the presence of methane and other volatile substances, including vinyl chloride. The Photovac 10A10 was used on-site to further determine the extent of contamination.

Using a gas-tight syringe, grab samples were taken from the emission stream of several vents. Samples of ambient air in and around the site were also taken. Several vinyl chloride standards were used to establish the relative retention times and to determine concentration. Concentrations of vinyl chloride in the vent emissions ranged from 10 to 50 ppm. Concentrations in the ambient air ranged from a few ppb to 100 ppb.

Laboratory analysis of the vent emissions corroborated field results. Vinyl chloride was not detected by laboratory analysis of the ambient air samples, although the detection limit for the method used (adsorption by activated charcoal followed by solvent desorption) is usually 1 ppm. The Photovac 10A10 therefore provided a more sensitive means of detecting vinyl chloride in air.

5.4 Air Monitoring Tubes - General

Air monitoring and sample collection can be accomplished using a system consisting of a portable air sampling pump equipped with one or a series of air sampling tubes. The pumps are calibrated by delivering a known volume of air through the sample tube per unit time. Determinations of which tube types to use are based on the types and suspected concentrations of chemical substances anticipated. A series of different sampling tubes is often used to maximise collection and monitoring efficiency. Two types of tubes

are most commonly used in environmental testing, namely detector tubes and sorbent tubes.

5.5 Air Monitoring Using Detector Tubes

5.5.1 Description. Detector tubes are glass tubes filled with a packing material treated with a chemical indicator. The detector tube is specific for the identification and semi-quantification of a chemical gas or vapour, depending on the chemical indicator used. When the sampled air contains that specific substance, a pre-determined colorimetric change occurs within the tube.²¹

The specific design of a detector tube determines the air sampling procedure. Some detector tubes are manufactured for use with a portable air-monitoring pump. The tube is attached to a calibrated pump, and a specific volume of air is pulled through the tube per unit time. Other detector tubes are manufactured for use with a calibrated plunger. Upon pulling the plunger back, a specific volume of air passes through the detector tube. This type of tube is manufactured in different sizes to allow for sampling of different fixed air volumes. Other types of tubes are available as personal monitoring devices to monitor worker exposure to hazardous vapours.¹⁶

Detector tubes are manufactured in the USA by National Draeger, Inc. of Pittsburgh, Pennsylvania ('Draeger tubes'), Matheson Gas Products of East Rutherford, New Jersey and Bendix/GASTEC of Lewisburgh, West Virginia. Detector tubes are not available for all gas types.

5.5.2 Evaluation. The utility of detector tubes is somewhat limited by:

- (i) their low accuracy (approximately $\pm 25\%$);
- (ii) tendency for leaks to occur during pumping of the air sample through the tube; and,
- (iii) the need for subjective reading of colour charts for determination of semi-quantitative results.

Despite these inherent limitations, detector tubes are easy to use, inexpensive, and can provide qualitative and semi-quantitative information. In this respect, they are useful as a supplement to more sensitive and accurate instrumental analysis techniques.

5.5.3 Application. Detector tubes may be used effectively as qualitative confirmation of the presence or absence of certain extremely toxic inorganic gases (ie H_2S , HCN , PH_3 , SO_2). They may also be used on-site to screen the headspace of containers.

Detector tubes are usually more useful however in a confined industrial workplace than at hazardous material sites. Interferences from other gases often present at hazardous incident sites tend to affect detector tube readings,²² usually resulting in higher concentrations being measured than are actually present.

5.6 Air Monitoring Using Sorbent Tubes

5.6.1 Description. Sorbent tubes are widely used for monitoring ambient air providing both qualitative and quantitative information. To collect a sample, a known volume of air is pumped through the tube. The contaminants in the sample are adsorbed and concentrated on the sorbent material in the sampling tube. The sorbent is then subjected to either thermal desorption or solvent extraction before conclusive analyses are performed. Two companies that manufacture sorbent tubes in the USA are Mine Safety Appliances Company (MSA) of Pittsburgh, Pennsylvania, and Matheson Gas Products.

5.6.2 Evaluation. Specificity in the use of sorbent tubes is dependent upon the sorbent medium with which the tube is filled, of which there are three principal categories:

- (i) porous polymers
- (ii) inorganic adsorbents
- (iii) activated charcoal and carbon.

The porous polymers most frequently used are Tenax GC, XAD resins, Porapak and Chromosorb series adsorbents. These polymers adsorb a wide range of compounds, including organophosphorous compounds, some hydrocarbons, organic nitrogen and sulphur compounds, inorganic acids, and vapours of certain pesticides. Once a sample has been collected, thermal desorption is commonly applied to remove these materials from the sorbent. Subsequently, qualitative and quantitative analyses are performed. When a porous polymer is required, Tenax is usually preferred as its high thermal limit (approximately 350°C) permits thermal desorption of the adsorbed compounds.²³ An additional advantage is that Tenax adsorbs organic materials of low molecular weight more efficiently than carbon at equal temperatures.

Problems associated with the use of solid sorbents include the irreversible adsorption of some compounds, limited retention capacity, sampling flow limitations, and atmospheric moisture content. Porapak and Chromosorb series adsorbents are particularly limited by their tendency to absorb water vapour during field use.

Inorganic adsorbents, such as alumina and silica gel, can be used to collect a wide range of contaminants based on their polarity. Silica gel is often used to collect haloacid gases, aromatic amines, and polar substances. However, field use of the inorganic adsorbents is limited by the tendency to retain water vapour, which reduces the number of active sites on the adsorbent medium.

Sorbent tubes containing activated charcoal or carbon will efficiently collect most high molecular weight organic compounds from an air sample, including benzene, carbon tetrachloride, chloroform, and xylenes. Once the sample has been obtained, the sorbent material is extracted with carbon disulphide and the extract is then analysed by an appropriate laboratory method. Activated charcoal and carbon tubes are very practical for use during field activities since their water absorption properties are significantly less than those of other sorbent tubes.

Sorbent tubes can be arranged in series to provide for maximum efficiency in the collection of chemical contaminants from an air sample. A simple arrangement of sorbent tubes might consist of a porous polymer tube containing Tenax followed by an activated carbon tube.

5.6.3 Application. The monitoring of ambient air using air monitoring tubes at contaminated sites frequently indicates the type and extent of chemical contamination present. Air monitoring serves as a preliminary investigation of a site prior to any large-scale investigation or sample collection, and it can be used to test the effectiveness of cleanup measures and remedial measures.

Abandoned Hazardous Waste Dumpsite, Pennsylvania. Air monitoring was performed by the US EPA at an abandoned hazardous waste dumpsite located in Pennsylvania in February 1983. As part of the air monitoring program, samples were collected from eleven on-site sampling stations, as well as from a nearby residential area. Sampling was accomplished using personal air sampling pumps equipped with air sampling tubes. Three different types of sorbent tubes were used; activated carbon tubes to collect organic vapours, silica gel to collect inorganic acids and aromatic amines, and Florisil to collect PCBs.

Chemical analysis of the sorbent tubes yielded qualitative and quantitative information regarding the levels and types of personnel protection required for workers operating at the site. The results also indicated the need for further sample collection at the site.

Abandoned Chemical Company, Texas. The ERT performed air monitoring operations at the site of an abandoned chemical company in Texas during August 1982. The ambient air sampling program was developed specifically to monitor for total organics, organic

solvents, and aromatic amines. Three types of sorbent tubes were used; activated carbon for organics; silica gel for aromatic amines and Tenax for the collection of total gas/vapours. Subsequent sample analyses revealed low level contamination from which recommendations were made regarding the appropriate protective levels for workers at the site

5.7 Other Air Monitoring Techniques

Air monitoring equipment in frequent use by the US EPA during hazardous material incidents and at waste sites was discussed in the preceding sections. Table 11.7 lists other instrumental monitoring techniques that may be used for air analyses. The compounds detected vary for each instrument, as does the instrument's sophistication and cost. Some of these instruments or kits may provide distinctive or supplemental information for on-site air monitoring.

6 WATER MONITORING EQUIPMENT

This section addresses the analysis of aqueous samples using a variety of portable field instruments and kits that permit rapid qualitative and semi-quantitative measurement of hazardous and toxic materials.

6.1 Toxicity Monitor

6.1.1 Description. The Beckman Microtox System²⁴ can be used to evaluate the acute toxicity of aqueous samples. The system consists of an analyser and chart recorder to measure the bioluminescence. There are four bottles of Microtox reagents:

- (i) bacteria (lyophilised photobacterium phosphorum);
- (ii) reconstitution solution to rehydrate and suspend the bacteria;
- (iii) diluent for preparing serial dilutions of the sample(s); and,
- (iv) osmotic adjustment solution.

To conduct the test, the bacteria are reconstituted and allowed to stabilise for fifteen minutes. Ten cuvettes are needed for each sample: nine for serial dilutions and one for a normalisation blank. The light emitted from the bacteria is measured before the addition of the sample, and at defined time intervals thereafter. From this data, the normalised percentage light decrease is determined and plotted against the concentration on semi-log paper to establish the concentration at which there is a 50% reduction in the light emitted

Table 11.7 Air Monitoring Techniques*

Instrument/Manufacturer	Deliverables	Other	Reference
Hydrogen Sulphide Analyser kit SKC, Inc, Eighty Four, PA	Hydrogen Sulphide	Tutwiler Principle, starch solution indicator	2
Model 715 Oxygen Monitor Beckman Instruments, Inc	Measures oxygen in gaseous samples or DO in aqueous or non-aqueous solutions	Battery powered system accuracy $\pm 1\%$ at given sample temperature $\pm 6\%$ for temperature variations	1
Instantaneous Vapour Detector Model 380 Sunshine Instruments, Co, Philadelphia, PA	Mercury vapours, acetone, aniline, benzene, benzyl alcohol, diethyl acetal, illuminating gas, naptha, pyridine, toluene, xylene	Includes battery-powered inverter unit, accuracy of $\pm 5\%$ of full scale 3.6 kg	3
Halide Detector GasTech, Mountain View, CA	Compounds containing halogens	Includes 3 calibration charts and a maintenance kit. 5.9 kg requires 120W 50/60 Hz. Accuracy: $\pm 5\%$ Precision: $\pm 3\%$ 15.9 kg requires 110W, 60 cycle AC Accuracy: $\pm 10\%$	1
Halide Meter Scott Aviation-Davis Instruments, Charlottesville, VA	Halogenated hydrocarbons in air particularly perchloroethylene, trichloroethylene, CCl_4 &	15.9 kg requires 110W, 60 cycle AC Accuracy: $\pm 10\%$	1
Calorimetric Analyser CEA Instruments, Inc	Acrylonitrile, ammonia, bromine, Cl_2 , formaldehyde, hydrazine, hydrogen chloride, hydrogen cyanide, hydrogen fluoride, hydrogen sulphide, methylhydrazine, nitrogen dioxide, oxides of nitrogen, phosgene, sulphur dioxide		3
Hiran-1A Foxboro Analytical, Inc Burlington, MA	Portable dispersive IR analyser, intended for quantitative analysis of multi-component mixtures	15.9 kg Precision and Accuracy: $\pm 2\%$	1.3
Model 511 Flame Ionisation Gas Chromatograph, Analytical Instrument Development, Inc Avondale, PA	Intended for air pollution and environmental health studies	Separate sample into individual components, concentration determination possible	3.1

Union POC-10 Portable Gas Chromatograph National Environmental Instruments, Inc. Warwick, RI	Developed for on-the-spot field analysis of trace gases in the ppm range	22 kg requires 115V, AC-60 Hz power source provided with a recorder, the Unicorder 20, TCD	1, 3
Custom made Multi-Parameter Test Kit for Air, Labette Chemical, Chesterton, IN	Amonia, Bromine CO, Cu, cyanide, hydrazine Lead, SO ₂ , H ₂ O, NO ₂	Kits are custom made to buyer's specifications	4
Ultra Gas-03S SO ₂ Analyser Calibrated Instruments, Inc. New York, NY	Sampling and analysis device for measuring SO ₂ in air. Needs same type of protection from adverse weather conditions	27 kg requires 100 Watts Detection limit: 0.005 ppm Interference by HCl, H ₂ S, H ₂ , H ₃ , not affected by CO ₂	1
Series 9000 Portable Gas Analyser System, Devco Engineering, Inc	Toxic gases or vapours in atmosphere or trace concentrations of contaminants in process streams	115V VAC, 60 Hz Accuracy: 2% at calibration point 5% over range	1
Series 2000 Ecolyser Portable Co Monitor, Energetic Sciences, Inc, Elmsford, NY	Measurement of CO in ranges from 0 to 3000 ppm	Self-contained rechargeable batteries, 4 kg. Precision and Accuracy: $\pm 1\%$ full scale	1
Model 03T Ozone Recorder Ozone Research and Equipment Corp. Phoenix, Arizona	Designed for ozone measurement	32 kg 110V, 400 Watts Accuracy: 3% of scale	1
Portable Flame Ionisation Meter Model 11-634 Scott Aviation/ Davis Instruments, Charlottesville, VA	Detects trace hydrocarbons in air, monitoring for toxic concentrations of solvents or process chemicals	13.6 kg range from ppm to volume %	1
Triton Beta-Gas Monitors, 1055B Johnston Laboratories, Inc Cockeysville, MD	Monitors radiogenic gases, to H ³ , C ¹⁴ , Kr, A, Rn	10.9 kg Accuracy: $\pm 10\%$ of full scale	1

References:

- 1 American Conference of Governmental Industrial Hygienists, 1978. Air sampling instruments for evaluation of atmospheric contaminants.
 - 2 SMC Catalogue and Guide to Air Sampling Standards, 1981.
 - 3 Cross, Harris, Lache, and Dillman, 1980. Instrumentation for detecting hazardous materials.
 - 4 Labette Chemical Products Co. Catalogue, 1983.
- * This tabular compilation of Air Monitoring Techniques is necessarily restricted to field portable instruments manufactured in the USA although similar kits and devices may be commercially available in other nations.

by the bacteria. This is known as the $EC_{50}(t,T)$, which refers to the effective concentration in terms of time (t) and temperature (T) two important parameters that must be noted when reporting results.

6.1.2 Evaluation. The Microtox System may offer a cost and time efficient alternative to bioassays such as the 96 hour fish toxicity test. The results have a statistical advantage over the bioassays of higher organisms owing to greater standardisation within the population and a sample population size exceeding one million. The relative toxicity data achieved with the Microtox System compares well with rat toxicity (correlation coefficient of 0.90) and fish toxicity (correlation coefficient of 1.0) studies for certain compounds.

According to Beckman Instruments, Inc.,²⁴ the major limitation of this technique is that it 'cannot fully define the toxicity of a particular substance in relation to other life forms and therefore results should be considered as an indicator of potential hazard'. In addition, coloration in samples requires an additional calculation during analysis. High turbidity can be accounted for by a correction factor if particulates are removed prior to analysis. There is also a 2% variability from geometric differences in sample cuvettes.

6.1.3 Application. The Microtox System was developed for monitoring waste treatment and industrial influent and effluent streams as well as toxic substances in the environment. The technique can also be used to study the synergistic and antagonistic effects of toxic mixtures and to determine the potential hazards of new chemicals. Microtox is not a chemical analysis method, and as such cannot identify or quantify toxicants. Hence, its major application is to determine the relative toxicity of contaminants present in an aqueous sample. In this regard, the Technical Assistance Team of the US EPA has used the Microtox for a variety of sample analyses and has found it useful for screening the relative toxicity of hazardous materials.

Greenville, Mississippi. Ethylene dichloride (EDC) leaked from a storage tank into an oxbow lake of the Mississippi River, settling at the bottom. Pumps were used to remove most of the EDC but the toxicity of the remaining residue required further determination. Samples were collected at various locations and the Microtox responses of the samples were compared to a dose-response calibration curve for EDC. The data indicated there were no signs of toxicity from any residual EDC present.

Stroudsburg, Pennsylvania. A tank containing penta-chlorophenol (PCP) contaminated oil leaked into the surrounding area. Sampling wells were drilled to recover the oil, but there was the possibility that the oil would migrate a nearby waterway. A retaining wall was built to stop further migration of the oil and samples were taken at

Table 11.8 Water Monitoring Techniques*

Instrument/Manufacturer	Deliverables	Other	Reference
Spectronix Mini-20 Bausch and Lomb Rochester, NY	Spectrophotometer w/ wavelength range of 400-800 nm	Battery operated, compact, easy to operate	1
Mini Conductivity Meter Hach Chemical Co. Loveland, CO	3 ranges: 0-100, 0-1000, 0-10000	0.7 kg, includes batteries and carry case	5
Model 16300 Conductivity Meter Hach Chemical Co. Loveland, CO	± 1% accuracy 5 ranges 0-2, 0-20, 0-200 0-2000, 0-20000	3.9 kg includes battery pack, ac	5
Model DB/1 Portable Colorimeter Hach Chemical Co. Loveland, CO	pH, 50 different water and wastewater tests	Batteries, AC	5
Model DB-EL/4 Portable Laboratory Hach Chemical Co. Loveland, CO	Spectrophotometer, titrator, cartridges		5
Model CYN-2 Cyanide Test Kit Hach Chemical Co. Loveland, CO	0-2.6 ppm free cyanide	Colorimetric test	6
Hazardous Materials Detector Kit Hach Chemical Co. Loveland, CO	pH, heavy metals, benzene, phenol, cyanide, conductivity, turbidity, nitrate, nitrogen, colour, sulphate, phosphate, NH_3 , fluoride	qualitative tests of non-specific nature to analyse for classes of	3 7
Mark XV Quality/Microprocessor Norton Instruments, Irvine, CA	temperature, salinity, conductivity, depth, pH, specific ions		2
Water Analysis Laboratory LaMotte Chemical, Chesterown, MD	alkalinity, NH_3 , Cl, Br, I, Chromium Copper, iron, F, nitrate, nitrite, phosphate (low), silica, sulphate, sulphide, pH, turbidity, TDS, conductivity, CO_2 , chlorine/salinity, DO		4
Luminescence Photometer Analytical Luminescence Lab, Inc San Diego, CA	bacterial biomass in sludge, toxic levels of waste, salt water constituents	1.1 kg maximum 100 samples/hr	2
8500 DO/DOB Meter, L C Heister, Co. Hillville, NJ	range of 0-20 ppm, LCD readout, for use in wastewater, BOD, Environmental monitoring applications		2
Custom Made Multi-Parameter Test Kit for Water, LaMotte Chemical, Chesterown, MD	amine, CO_2 , cyanide, formaldehyde, heavy metals, iodine, Fe, Pb, phenol	Kits are custom made to buyers' specifications	4
Cam-4, Midwest Research Institute, Kansas City, MO	monitor the levels of organophosphate and carbamate pesticides in water	12V DC maximum of 8 continuous hours 110V AC 13.6 kg	8

References:

- 1 Snyder, Tomkin and McKisick, 1980. Development of hazardous/toxic wastes analytical screening procedures.
- 2 Pollution Equipment News, April, 1983.
- 3 Crose, Harris, Lachs, and Dillman, 1980. Instrumentation for detecting hazardous materials.
- 4 LaMotte Chemical Products Co. Catalog, 1983.
- 5 Hach Chemical Co. Products for Analysis, 1980.
- 6 Hach Chemical Co. Water Test Kits, 1971.
- 7 US EPA, 1982. Environmental emergency response unit capability.
- 8 US EPA, 1980. Cam-4, A portable warning device for organophosphate hazardous material spills.

* This tabular compilation of water monitoring techniques is necessarily restricted to field portable instruments manufactured in the USA although similar kits and devices may be commercially available in other nations.

wells known to contain the oil and at various other areas of suspected leaching. Microtox analysis confirmed the presence of PCP in known wells and demonstrated that the retaining wall had halted migration of the oil.

Laskin's Waste Oil Service, Jefferson, Ohio. At a waste oil service site in Jefferson, Ohio, a number of ponds and lagoons contained an oil layer laden with polychlorinated biphenyls and other hazardous materials. A mobile carbon treatment unit was used to pump the contents of the lagoon through activated charcoal to remove contaminants, and the Microtox System was used to monitor the toxicity of the effluent as the cleanup progressed.

6.2 Water Monitoring Techniques

Field test instruments and kits that could be applied to the analysis of water (and possibly soil) samples are listed in Table 11.8. Although these kits have not been used or evaluated by the US EPA to date, they may serve to provide additional field monitoring capabilities. The majority of these kits are designed to measure specific contaminants in aqueous solution and thereby facilitate rapid qualitative and semi-quantitative measurements of on-site and surrounding area water samples. Analysis of surrounding area waters can indicate any natural background conditions which may possibly interfere with on-site analyses. It may also be feasible to use these kits to survey contaminated land by simply analysing aqueous extracts of oil/sediment. However, these procedures are currently unverified, and the methods will require further validation.

7 SOIL MONITORING TECHNIQUES

The complex composition of soil with its inherent inorganic and organic background, and differential ability to sorb and desorb contaminants, makes soil analyses difficult. There are relatively few simplified procedures, techniques or kits available that can analyse contaminated soil directly. Almost all current procedures incorporate an extraction step (eg Soxhlet or acid digestion) and sample cleanup. However, by performing a rapid aqueous extraction of the soil it may be possible in many instances to use kits and techniques that normally measure contaminants in water. In some instances, an aqueous extract of soil might be a better indicator of the relative 'bioavail-ability' of a particular contaminant. If a contaminant is bound so strongly to the soil that it will not desorb into water, then it may not be readily available to exert acute toxic effects. The applicability of a kit or technique to analyse an aqueous soil extract should be evaluated before actual field use to verify that background soil constituents do not interfere with the analysis.

Table 11.9 Soil Monitoring Techniques*

Instrument/Manufacturer	Deliverables	Other	Reference
Custom Made Multi-Parameter Test Kit for Soil, LaMotte Chemical, Chestertown, MD	nitrate, phosphorous, K, Ca-Mg, organic matter, boron, cobalt, Cu, zinc, pH, dissolved salts dissolved solids	Kits are custom made to buyers' specifications	1
Soil Test Laboratory Model 1700 Hach Chemical Co. Loveland, CO	soil nutrient, pH, ammonia, nitrate, N, lime, phosphorous	includes a battery operated photo-electric colorimeter and built in pH meter	2
Soil Extraction Kit Hach Chemical Co. Loveland, CO	used to prepare soil sample extracts for testing with water test kits		2

References:

- 1 LaMotte Chemical Co. Catalog, 1983.
- 2 Hach Chemical Co. Products for Analysis, 1982.

* This tabular compilation of Soil Monitoring Techniques is necessarily restricted to field portable instruments manufactured in the USA although similar kits and devices may be commercially available in other nations.

A relatively new instrument which was developed for field use is the portable X-ray fluorescence unit. X-ray fluorescence is a commonly used laboratory technique for determining the composition of samples. An X-ray source irradiates the sample, causing the sample to emit X-rays at various energies characteristic of the chemical elements in the sample. A spectrometer measures the energy levels of the X-rays emitted to analyse the sample composition. Several versions of these units are available from Martin-Marietta, Inc and Texas Nuclear Division, Ramsey Engineering Company. The manufacturers anticipate that these portable instruments should detect elements in concentrations as low as 10 ppb. This technique might also be used to detect fixed organochlorine compounds (ie PCBs) and organo-metallics (ie tetraethyl lead, arsenates) in soil samples.

Simple soil tests that yield additional information about soil conditions are also commercially available in the USA. Several soil analysis kits that are relatively inexpensive and simple to use are listed in Table 11.9. These kits yield background information on critical soil parameters which ultimately determine the habitability of the soil in conjunction with the presence or absence of toxic contaminants. In addition, Sections 4, 6, and 9 describe techniques and equipment which may be applicable for soil analyses with appropriate modifications. The most applicable techniques are those that measure contaminants directly in water (ie aqueous soil extracts). Selection of the appropriate technique may also be influenced by the types and quantities of contaminants present and the ability of the technique to adequately resolve the analysis.

8 SPECIALISED SCREENING TECHNIQUES

Sampling techniques, compatibility tests, and air, water and soil monitoring equipment have been discussed above. Other techniques, employed in special situations requiring the screening for particular contaminants, are the subject of this section, which addresses two problems of frequent concern namely, determination of the presence and abundance of (i) phenolic materials and (ii) polychlorinated biphenyls (PCBs).

8.1 Total Phenol Analysis

8.1.1 Description. Aqueous phenolics can be quantified spectrophotometrically using 3-methyl-2-benzothiazolinone hydrazone (MBTH) as a coupling agent. The method described here adapts EPA Method 420.3 (Methods for Chemical Analysis of Water and Wastes) to the requirements of on-site analysis. The major modification to Method 420.3 is exclusion of the distillation process. The distillation is included in Method 420.3 to remove interferences, but with sufficiently clean samples, the distillation step can be omitted.

8.1.2 Evaluation. Although the MBTH spectrophotometric method uses a large number of prepared solutions, it can be a valuable on-site method of analysis. A portable spectro-photometer (such as the Bausch and Lomb spectronic mini 20) and a portable pH meter (or pH paper) can be used. When many samples require testing, an assembly line approach can yield an analysis rate of approximately 10 samples per hour.

An inherent inaccuracy in the method results from quantifying the samples on a phenol calibration curve. Many phenolic wastes contain a variety of phenols which do not have the same colour response to MBTH coupling. When this mixture of phenols is quantified on the phenol calibration curve, the results will represent the minimum concentration of phenolic compounds present. Although it is not possible to differentiate between the various types of phenols, the method is capable of measuring phenolic materials from 50 to 100 ug/l in water. There is a concentration/extraction step with chloroform which can increase the sensitivity of the technique to 2 ug/l.

8.1.3 Application. Most phenols exhibit some toxicity to humans and eleven phenolic compounds are considered priority pollutants by the US EPA. Phenols are often an indication that other pollutants are present. This method can be used to analyse for phenolic compounds in drinking, surface and saline waters, and in domestic and industrial wastes. However, the method is limited to quantifying the minimum concentration of phenolic compounds in a mixture.

Laskin's Waste Oil Service Site, Jefferson, Ohio. The ERT used this method at the Laskin's Waste Oil Service site in Jefferson, Ohio. A number of ponds and lagoons on the site contained an oil layer (laden with polychlorinated biphenyls and other hazardous materials) and a water layer. After the initial characterisation of the water indicated the presence of phenolic compounds, the ERT used the MBTH spectrophotometric method (without distillation or concentration) to monitor the influent and effluent streams of a mobile carbon treatment unit. The analyst was able to quantify ten samples per hour and experienced no significant problems with the on-site methodology.

8.2 Polychlorinated Biphenyl Field Test Kit

8.2.1 Description. The Centec Analytical Services TR 201 is a lightweight, battery operated, fully portable field testing kit designed for semi-quantitative analysis of PCBs in transformer fluid.^{25,26} To analyse PCBs, chlorine atoms are chemically stripped from the biphenyl rings and analysed electronically with an ion selective probe. A digital display in millivolts corresponds to the concentration of PCBs present. This analysis is a three step

procedure and averages five minutes per sample. The kit is calibrated at the start of the analysis and hourly thereafter. It must also be calibrated each time it is moved to a new location.

8.2.2 Evaluation. Major advantages of the PCB field test kit are: (i) that it does not require the skills of a chemist to perform the analysis or to interpret the data, and (ii) it is a cost-effective technique (ie approximately 90% less expensive than GC-ECD analysis). In laboratory studies using known Aroclor standards, the results of the TR 201 and GC-ECD analysis were comparable (ie correlation coefficient of 0.96 at levels above 7 ppm). The company claims a precision of $\pm 5\%$ and accuracy of $\pm 10\%$ in the analysis of PCB-contaminated oil. However, with unknown Aroclor samples, the accuracy of the method drops to $\pm 17\%$ as a result of the use of an average percentage Aroclor standard for instrument calibration. Consequently, samples approaching the disposal category limits of 50 and 500 ppm (see below) require quantitative analysis by gas chromatography.²⁵

The kit's major limitation is that it cannot identify or quantify PCBs in soil or water matrices, and the presence of certain solvents (eg alcohols) negates the chlorine stripping reaction. In addition, the ion selective probe cannot differentiate between chlorine from PCBs or from other chlorinated compounds such as pesticides. The major use for the kit is to analyse oil samples where the presence of PCBs is expected, and when other chlorinated compounds are not present to cause interferences. However, the kit may be used as a screening tool for qualitatively determining the presence and approximate concentration of chlorinated compounds contained in drums at hazardous waste sites.

8.2.3 Application. The US EPA has taken regulatory action to prohibit the resale, distribution, and manufacture of PCBs after established deadline dates, and has restricted their disposal and handling. The following criteria were set to categorise the dielectric fluid in transformers, the primary source of PCB usage:

Greater than 500 ppm PCBs:	PCB transformers
50-500 ppm PCBs:	PCB contaminated transformers
Less than 50 ppm PCBs:	Non-PCB transformers
PCBs not detected:	No restrictions

The Centec Model TR 201 PCB Field Kit is useful for the mandated screening of fluids in transformers requiring repairs and in the inspection of transformers that are 'critically located' (US Federal law requires the removal of any transformer categorised as a PCB transformer or PCB contaminated transformer located near water supplies, crops, or livestock.) Quantification of PCBs in transformer fluids is essential in determining the appropriate disposal methods to comply with federal standards and restrictions,

since any untested transformer is considered to be a PCB transformer unless proven otherwise. The kit is also useful in the on-site analysis of transformer content spills.²⁶ The primary users of the TR 201 are utility companies and transformer repair companies.

9 MOBILE ANALYTICAL LABORATORIES

The increasing number of hazardous material spills and the continuing discovery of abandoned chemical waste disposal and other contaminated sites requires both rapid and effective methods for on-site chemical analysis. Immediate analytical data is often needed to:

- (i) assess the extent of environmental contamination;
- (ii) make decisions regarding methods of cleanup and disposal; and,
- (iii) efficiently monitor ensuing cleanup efforts.

The use of mobile analytical laboratories effectively accomplishes this by eliminating the delay associated with the transport of samples to distant laboratories for comprehensive analysis.

As noted in the preceding sections of this Chapter, the utility of field test kits and portable instruments is somewhat limited in that these techniques do not afford a complete qualitative and quantitative characterisation of the contaminants present in a particular matrix. Mobile laboratories on the other hand provide for: (i) versatile capabilities based on the array of instruments available; (ii) rapid sample turn-around time and cost-effective analyses; and (iii) accurate qualitative and quantitative multicomponent analysis.

9.1 EPA Mobile Laboratory

9.1.1 Description. The US EPA's Oil and Hazardous Materials Spills (OHMS) Branch and Environmental Response Team (ERT) operate a mobile laboratory which provides rapid on-site analytical services in support of multimedia hazardous waste cleanup activities. Housed in a 35 foot (10.7 m) semitrailer van, the mobile laboratory is equipped with numerous analytical instruments, including:

- (i) two computerised gas chromatographs equipped with flame ionisation, electron capture, and nitrogen-phosphorus detectors;
- (ii) a computerised gas chromatograph/mass spectrometer;

- (iii) an emission spectrometer;
- (iv) infrared and fluorescence spectrophotometers; and
- (v) a total organic carbon analyser.

This broad range of analytical capabilities permits accurate chemical analysis of most organic and inorganic substances encountered at hazardous waste sites.²⁷ For the safety of laboratory personnel, the mobile laboratory is furnished with glove boxes, a fume hood, vented solvent locker, explosion-proof refrigerator, fire blanket, eye wash, and safety shower. The heating and air conditioning system is designed to provide totally non-recirculated air.

The mobile laboratory is readily dispatched to any part of the United States within a few hours of an official request for emergency analytical services. The laboratory is staffed with two to four chemists, depending on the type and number of analyses requested. Contact is generally maintained with technical co-ordinators in the central laboratory in New Jersey, through the use of telephone lines and telefacsimile units.²⁸

9.1.2 Evaluation. Specialised rapid and direct methods of analysis are used in the mobile laboratory that avoid time consuming and tedious extraction, evaporation, concentration, and chromatographic cleanup steps and enable rapid processing of numerous samples during field use.

Sample preparation procedures. Several rapid, cost effective sample preparation and pretreatment procedures were developed for use in the mobile laboratory that require a minimum amount of space. Rapid extraction methods have been developed for a variety of hazardous materials and petroleum oils in aqueous media. A single step extraction is used, in which a small amount of solvent is added directly to the water sample and the mixture is agitated with a magnetic stirrer for 30 minutes. The layers are permitted to separate, and a portion of the organic phase is removed for analysis. The use of a single step extraction procedure also reduces potential contamination from multi-step extractions.

Contaminated soil and sediment samples are extracted by using a single step mechanical shaking technique. The extraction solvent is added to an erlenmeyer flask containing a weighed sediment sample which is then placed on a gyratory device for approximately 20 minutes. This rapid extraction procedure significantly reduces sample analysis time when compared with conventional methods incorporating exhaustive soxhlet and alcoholic-KOH extraction procedures.

Table 11.10 Selection of Chromatographic Cartridges

Compounds	Recommended Chromatographic Cartridge	Precautions & Limitations
Pesticides	Florisil	Extracted phenols are derivatized with penta-fluorobenzylbromide (PFB) before the extract is cleaned with silica gel.
PCBs	Florisil	
Phenols	Silica gel	N-Nitrosodiphenylamine decomposes in the GC injection port and is detected as diphenylamine. Therefore all diphenylamine in the sample must be removed by Florisil to prevent interference.
Phthalate	Florisil or	
Esters	Silica gel	
N-Nitrosodium phenylamine		
Organochlorine	Florisil	The extract may be cleaned with Florisil to prevent phthalate interference.
Pesticides	Florisil	
Haloethers	Florisil	Because of the extreme toxicity of this compound the analysis method should be reviewed thoroughly before it is used.
Chlorinated	Florisil	
Hydrocarbons		
2,3,7,8 - Tetra	Silica Gel	
chlorodibenzo-	cartridge	
p-dioxin	followed by an	
(TCDD)	Alumina cartridge	
Herbacides	Florisil	
Aflatoxins	Florisil	
Mycotoxins	Florisil	
Lipids	Florisil	
Alkaloids	Florisil	
Steroids	Florisil	
Benzidine	Florisil or	
Diphenylhydrazine	Silica gel	
	C18	
Toluene	C18	Pre-rinse cartridge with distilled water and elute with methanol.
Nitrosamines	Florisil	

A sample consolidation procedure is employed when a large number of drummed chemical wastes are analysed. Since complete characterisation of the contents of each drum is neither practical nor cost efficient, compatible waste samples are composited, in order to decrease the number of analyses.²⁹

Environmental sample cleanup is facilitated by the use of SEP-PAKTM chromatographic cartridges which simplify sample preparation procedures by eliminating the need for column and separatory funnel extractions. Sample extracts are injected onto a cartridge, followed by a known amount of solvent, to selectively isolate (elute or retain) components of interest from interferences. SEP-PAKsTM are available containing alumina, C₁₈, silical gel, or Florisil packing material. Table 11.10 provides guidelines for selecting the appropriate sorbent material for a particular class of compounds.

Instrumental analysis procedures. The analytical methodology used aboard the mobile laboratory specifically addresses the analysis of US EPA designated priority pollutants. These compounds comprise a broad range of hazardous materials including pesticides, polychlorinated biphenyls, base neutrals, phenolics, purgeables, and heavy metals. The priority pollutants represent a template of substances potentially encountered at contaminated sites. The methodology employed permits accurate quantification of these materials down to the low part per billion level. Table 11.11 provides a listing of the analytical methodology developed by the OHMS Branch.

The methods used most frequently employ gas chromatographic techniques. The gas chromatograph provides several capabilities for the quantitative determination of organic priority pollutants in air, water, sediments, and the contents of chemical storage tanks and drums. Quantification of sample extracts containing pesticides, polychlorinated biphenyls, and base neutrals is accomplished through the use of a mixed phase column consisting of 1.5% SP-2250 and 1.95% SP-2401 coated on 100/120 mesh Supelcoport using electron capture detection (ECD). Sample extracts containing phenolic compounds are analysed using a column containing 1% SP-1240 DA on 100/120 mesh Supelcoport and a flame ionisation detector. Purgeable priority pollutants are analysed using a purge and trap sampler, a column packed with 1% SP-1000 on 60/80 mesh Carboxen B, and a flame ionisation detector. Automatic samplers on the gas chromatographs permit continuous overnight operation thereby facilitating rapid sample throughout.

The gas chromatographic techniques employed by the mobile laboratory are augmented through the use of computerised gas chromatography/mass spectrometry. This enables separation of complex mixtures into their individual constituents for compound identification. Interpretation of mass spectra is accomplished by internal library searching and matching for priority pollutant

Table 11.11 OHMSB Chemical Analysis Methods and Special Techniques (from Gruenfeld et al³⁰)

A GAS CHROMATOGRAPHY

- 1 Rapid quantification of pesticide mixtures in water
- 2 Rapid quantification of polychlorinated biphenyls in water*
- 3 Rapid quantification of basic and neutral materials in water
- 4 Rapid quantification of polychlorinated biphenyls in sediments
- 5 Rapid extraction of oils from sediments for GC profile analysis

B FLUORESCENCE SPECTROSCOPY

- 1 Rapid quantification of hazardous materials directly in water
- 2 Rapid quantification of oil directly in water
- 3 Rapid quantification of fluorescing organics in water
- 4 Rapid quantification of hazardous materials in sediments
- 5 Rapid quantification of oils in sediments

C INFRARED SPECTROSCOPY

- 1 Rapid quantification of water dispersed petroleum oils
- 2 Rapid quantification of petroleum oils in sediments
- 3 Silica gel treatment for the separation of hetero-organics from hydrocarbons

D SAMPLE PREPARATION PROCEDURES

- 1 Weathering small amounts of oil
- 2 Removal of sulphur from sediment extracts

E SAMPLE ANALYSIS PROTOCOL

F QUALITY ASSURANCE/QUALITY CONTROL

- 1 Chemistry staff quality assurance program
 - 2 Protocol for preparing synthetic samples of hazardous materials in water
 - 3 Protocol for preparing synthetic samples of hazardous materials and petroleum oils in sediment
-

identification. Files of spectra are maintained on disks to facilitate retrieval and storage.

Cost effective analysis of inorganic compounds is performed by plasma emission spectrometry. Emission spectrometry enables analysis of over 70 elements in complex or concentrated environmental samples, with reduced sample preparation. A multi-channel optical cassette containing 20 pre-selected exit slits (including the priority pollutant metals), provides the capability to analyse for 20 elements simultaneously. After calibration of the instrument, one sample can be analysed every 2 minutes with the concentration of 20 elements displayed on a printer.

Synchronous excitation (SE) fluorescence spectroscopy is also used in the mobile laboratory for the analysis of organic priority pollutants and petroleum oils. The development²⁹ of several analytical methods based upon the SE fluorescence technique has considerably reduced sampling preparation and analysis time.^{30,31,32} Direct in-situ quantification of hazardous materials in aqueous media enables detection of fluorescing organics at the part-per-billion level.³³

Infrared spectroscopy is almost exclusively employed for the identification and quantification of petroleum oils extracted from various matrices. Several methods have been developed to rapidly extract and quantify petroleum oils in water and sediments.^{34,35} An unusually time efficient method for analysing petroleum oils in sediments entails extracting the samples with carbon tetrachloride via gyratory agitation. The extract are then diluted for infrared spectroscopic measurement which provides accurate quantification of petroleum oil in the sediment.

Total organic carbon (TOC) analysis is a useful technique for measuring the total organic content of an aqueous sample. The US EPA has employed TOC analysis in conjunction with a mobile carbon treatment unit designed to remove organic contaminants from aqueous media. TOC measurements of influent and effluent samples facilitate on-site monitoring of the cleanup and removal efficiency of organics from large bodies of water.

9.1.3 Application. Since its development in 1976, the EPA mobile laboratory has responded to 19 emergencies (see Table 11.12). Examples of three emergency responses are presented below.

Niagara, New York. Perhaps the most noted incident in which the rapid extraction methods were applied was the emergency response activity conducted near Niagara, New York, in October 1978. Over 200 families living along the abandoned Love Canal waste disposal site had to permanently evacuate their homes when toxic chemicals seeped through the ground into their basements

Table 11.12 EPA Mobile Laboratory Emergency Response Actions

Location of incident	Date	Compound(s) analysed	Technique
Haverford, Pennsylvania	Nov 1976	Pentachlorophenol; Oil	GC; IR
Dittmer, Missouri	Apr 1977	Oil, PCBs	GC
Kernsville, N Carolina	Jun 1977	Oil, mixed chemicals	FS
Oswego, New York	Oct 1977	PCBs	GS
Niagra, New York	Oct 1978	Chlorinated benzenes, PCBs, hexachlorocyclohexanes (BHC)	GC
Pittston, Pennsylvania	Aug 1979	c-dichlorobenzen	GC
Sharptown, Maryland	Feb 1980	Oil	IR
Crosby, Texas	Feb 1980	PCBs; oil	GC; IR
Manassas, Virginia	Mar 1980	Total fluorescing organics	FS
Elizabeth, New Jersey	Apr 1980	PCBs, o-dichlorobenzene	GC
Kingston, New Hampshire	Jan 1981	Volatile organics, PCBs	GC;
		Chlorinated pesticides;	Redox
		Oxidation-reduction potentials	kit
La Marque, Texas	Sep 1981	1,2-dichloroethane, 1,1,2-trichloroethane	GC
Fort Lawn, S Carolina	Oct 1981	PCBs, chlorinated pesticides	GC
Kingston, New Hampshire	Nov 1981	PCBs	GC
Old Forge, Pennsylvania	Apr 1982	PCBs	GC
Epping, New Hampshire	Apr 1982	Volatile organics; Total organic carbon	GC; TOC
Rock Creek, Jefferson, Ohio	Sep 1982	Volatile organics	GC
Epping, New Hampshire	Dec 1982	Volatile organics	GC
Kent, Washington	Apr 1983	Volatile organics, PCBs, heavy metals	GC; ES

Key: ES = emission spectroscopy
 FS = fluorescence spectroscopy
 GC = gas chromatography
 IR = infrared spectrophotometry

and gardens. Included with the more than 20 000 metric tonnes of industrial wastes in the Canal was an estimated 300 different toxic chemicals, nearly 10% of which were classified as mutagens, teratogens, or carcinogens. The mobile laboratory was assigned to analyse leachate and sediment samples for priority

pollutants. The samples were extracted according to the rapid extraction techniques developed for the analysis of base neutrals, phenolics, and PCBs. Gas chromatographic analysis of the extracts revealed the presence of chlorinated benzenes, hexachlorocyclohexanes (BHC), and PCBs.

Manassas, Virginia. In March 1980, a massive quantity of kerosene spilled into a small river leading to the Occoquan Reservoir, threatening the drinking water supply of Manassas, Virginia. Water samples were analysed aboard the mobile laboratory to monitor the concentration of total fluorescing organics. The samples were cooled to 4°C to prevent volatility losses and then rapidly extracted with cyclohexane using a spinning technique. The extraction step was incorporated into the method to avoid interferences encountered during direct in-situ measurements of the river water. The extracts were then quantified by synchronous excitation fluorescence spectroscopy.

Epping, New Hampshire. An emergency cleanup activity utilising a carbon treatment unit was conducted in March 1982. A contaminated solvent lagoon located in Epping, New Hampshire was in danger of overflowing. The water from the lagoon was pumped through a carbon treatment unit and discharged into holding tanks. The mobile laboratory monitored the cleanup efficiency of the unit by analysing influent and effluent samples for purgeable priority pollutants and total organic content.

9.2 Mobile MS/GC System

9.2.1 Description. The TAGA™ (Trace Atmospheric Gas Analyser) 6000 Mobile Mass Spectrometer System, manufactured by Sciex, Inc, is a self-contained computerised mobile laboratory providing real time air monitoring for the analysis of trace levels of organic and inorganic pollutants. The laboratory is equipped with a tandem mass spectrometer capable of providing both identification and quantification of contaminants in the atmosphere with minimal sample preparation.

The system is designed to monitor low levels of contaminants in air under rapidly changing conditions. As the laboratory travels through a contaminated area it continuously monitors the ambient atmosphere by drawing air through a sample probe mounted on the roof of the vehicle. The high sampling flow rate minimises memory effects, resulting in a rapid response to concentration changes at the sample inlet. The computer provides concentration contour maps of hazardous material plumes. Figure 11.5 illustrates the plume of aniline concentrations in the atmosphere near a chemical plant as seven transits of a downwind area were made during a twenty minute period.

effects, resulting in a rapid response to concentration changes at the sample inlet. The computer provides concentration contour maps of hazardous material plumes. Figure 11.5 illustrates the plume of aniline concentrations in the atmosphere near a chemical plant as seven transits of a downwind area were made during a twenty minute period.

9.2.2 Evaluation. Although the TAGATM 6000 system is expensive, the manufacturer claims that the system's flexibility, reliability, sensitivity, and rapid analysis capability provide exceptionally cost effective analyses. Hazardous waste site

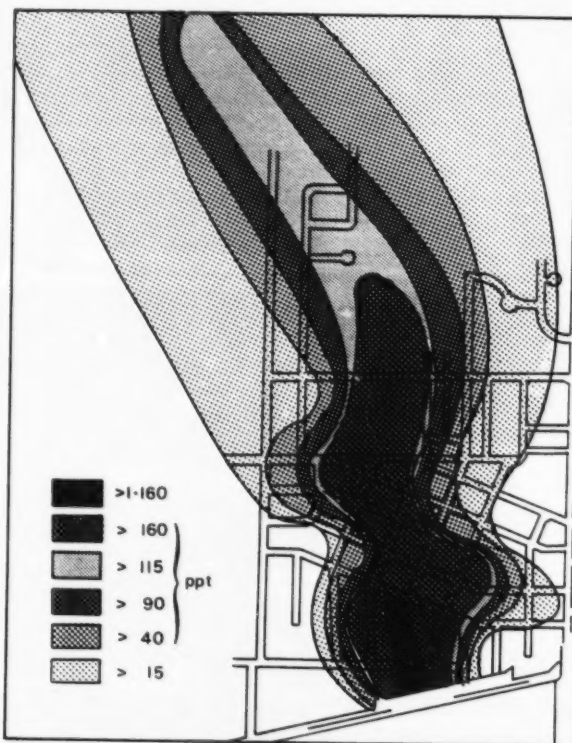


Figure 11.5 Plume Contour Plot of Aniline Concentrations in Atmosphere Near Chemical Plant as Measured by the TAGA 6000 Mobile Mass Spectrometer System (after Karasek³⁹)

investigations are normally conducted by on-site sampling followed by off-site laboratory analysis, creating a delay of one to two weeks for results. This delay is eliminated by the real time monitoring capability provided by the TAGATM system.

9.2.3 Application. The scope of applications for the TAGATM 6000 in environmental areas is broad. It has been used in emergency monitoring of toxic spills and in investigating dump sites. Special probes allow the screening of soil samples to determine the extent of contamination at a particular site. Air monitoring can be performed continuously during the cleanup of a site, providing additional safety measures for on-site workers. Because direct sampling of smokestack emissions is possible, the unit can also be used for monitoring combustion by-products. Several examples citing the use of the TAGATM 6000 are presented below.

Niagara, New York. In October of 1978, the TAGATM 6000 was employed by the Canadian Environmental Protection Service and New York State officials to develop information on the location of buried drums of hazardous waste that were leaching into the ground at Love Canal. The system was used as a rapid means of identification of contaminants present and as an alarm system to alert workers to dangerous levels of chemical releases into the immediate atmosphere surrounding the excavated areas.

Saskatchewan, Canada. In July 1979, Sciex responded to an accidental underground pipe leak in Saskatchewan, where PCBs were spilled. The TAGATM 6000 was able to analyse the surrounding soil to determine the extent of the spill and the concentration of PCBs present.

Mississauga, Ontario. When a train carrying chlorine gas and propane gas derailed in Ontario, the TAGATM 6000 traversed streets in a wide area downwind of the wreckage. Chlorine concentration profiles were developed to help officials decide which areas should be evacuated. The burning of propane resulting from the crash, in combination with the chlorine gas, had the potential to react to form deadly halogenated hydrocarbons. The TAGATM 6000 analysed for these compounds during cleanup efforts, and checked for remaining chlorine pockets before allowing re-entry into evacuated areas (see Figure 11.6).

10 CONCLUSIONS

The Pilot Study Groups' definition of contaminated land and classification of the hazards and problems associated with contaminated land were presented in Chapter 1. Contaminated land was defined as land where toxic substances are present that in sufficient

quantities or concentration pose a threat to the health and safety of users or occupiers of land or of workers engaged in its redevelopment or reclamation. Numerous analytical methods have been developed over the past decade in response to the increased need for rapid on-site assessment of contaminated land. Rapid on-site methods of chemical analysis facilitate monitoring of toxic and hazardous substances and thereby minimise exposure of potential targets (ie, on-scene personnel, site users, etc) to these hazards. In addition, by monitoring the general level of contamination at a particular site, these analytical techniques assist on-scene co-ordinators in identifying unacceptable and unhealthy conditions that may require immediate remedial response.

A large number of analytical techniques that are currently in use by the US Environmental Protection Agency and its private contractors for monitoring contaminated sites in support of cleanup

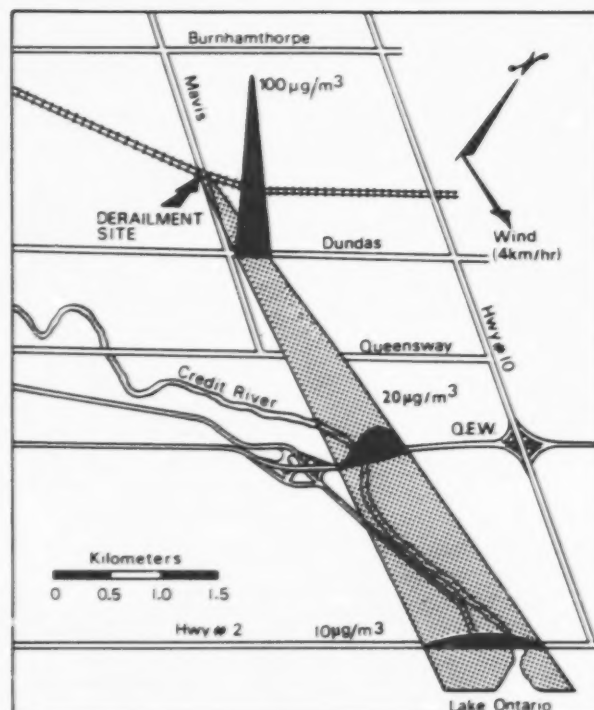


Figure 11.6 Three Plume Cross Sections taken by the TAGATM 6000 Mobile Mass Spectrometer System Following a Chlorine Spill (after Lane and Thomson⁴⁰)

and reclamation efforts have been reviewed in this Chapter. The reader is advised that the techniques described herein represent a user survey and are not meant to be all-encompassing, since other techniques are either commercially available or being developed as research programmes progress. The methods surveyed vary in complexity, ranging from non-specific, semi-quantitative screening techniques to compound specific, quantitative, multi-parametric protocols. These methods are extremely useful when applied to the initial appraisal of chemical waste sites and for monitoring the general levels of contamination at a particular site. Major advantages realised through the use of such field-oriented methodology are real time sample analyses and reduced costs of site investigations. Disadvantages include the limitations inherent in key analytical parameters (ie, accuracy, selectivity, sensitivity, etc) when portable instruments and field test kits and devices are employed.

The ultimate choice of on-site techniques for evaluating contaminants will be determined by many factors. Typical decision criteria include:

- (i) the type and size of the site and associated environmental factors;
- (ii) the types and abundances of contaminants anticipated at the site; and
- (iii) the level of effort required at the site (ie reconnaissance, preliminary and full surveillance, monitoring, and remediation and follow up activities). These efforts are further affected by the intensity of response required (ie spill and emergency response vs reclamation efforts).

There is a continuing need for the development of new techniques and refinement of others that can provide on-site analyses for the vast array of hazardous substances and sample matrices (ie, sludges, concentrated wastes, etc) encountered at contaminated sites. Application of practical chemical analysis methodology lends valuable support to management decisions involving the mitigation of immediate and long-term environmental hazards, particularly recommendations regarding appropriate remedial measures and ensuing reclamation efforts.

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